

TITRIMETRIC PROCEDURES

When a laboratory receives a new lot of commercially prepared titrant(s), or prepares a new batch of titrant(s), it must be standardized prior to use.

Reagent standardizations should be performed and recorded on a monthly basis (for most reagents). If sample analyses are performed less frequently than once a month, then check Normality at beginning of each analysis. Some methods require standardizations prior to each analysis, check method for requirements.

Reagent Logbook needs to include:

- a) reagent
- b) procedure for making reagent in-house. All weights and volumes must be specified: vol. (ml), weight (g)
- c) lot number of reagents commercially purchased
- d) calculation formula and actual calculations used to find Normality of reagent
- e) date
- f) analyst
- g) normality

Reagent bottle labelled with:

- a) reagent
- b) date opened (prepared)
- c) Normality checked (value)
- d) analyst
- e) expiration date (either manufacturer's or if prepared, check method)

Titrant reagents have different shelf-lives and storage conditions:

ex: Magnesium EDTA - maximum shelf-life of one month.

Discard sooner if 1 - 2 ml added to sample fails to produce a pH of 10.0 ± 0.1 at end point of titration. Store in tightly stoppered plastic bottle. Read each method to learn reagent shelf-life and storage conditions.

Potentiometric titrant recording requirements in **Sample Analysis Records**:

- a) initial pH (prior to titrating)
- b) final pH (after titrating)
- c) intermediate pH reading (in some methods - ex: alkalinity, in low concentrations)

Duplicates need to be performed at a rate of ten percent. If method requires digestion, a sample spike may also be needed - check method for requirements.

Check method to see if reagent blank analysis is required.

Always specify weights and volumes.

Some primary standard solutions (NIST certified) do not have to be standardized on a monthly basis. Check the NIST Special Publication 260 for specific information.